

[54] **FRICTION REDUCING AGENT FOR LUBRICANTS**

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[51] Int. Cl. **C10m 1/46**

[58] Field of Search **252/32.7 E, 75, 78, 32.5**

[56] **References Cited**

UNITED STATES PATENTS

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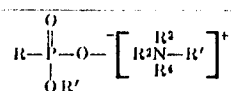
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[57] **ABSTRACT**

Ammonium salt of alkyl alkanephosphonates represented by the formula:



in which R is a substantially straight chain aliphatic radical having from about 11 to 40 carbon atoms, R' is a lower aliphatic radical having from one to eight carbon atoms, R² is a hydrocarbyl radical having from 1 to 40 carbon atoms and R³ and R⁴ are hydrogen, a hydrocarbyl radical having from one to 40 carbon atoms, or a substituted hydrocarbyl radical having amino, alkylamino or hydroxyl functional groups, the reaction of preparing same from an ester of an alkane-phosphonic acid, and a lubricating oil composition containing an ammonium alkyl alkanephosphonate.

5 Claims, No Drawings

FRICION REDUCING AGENT FOR LUBRICANTS

BACKGROUND OF THE INVENTION

1. Field of the Invention

The demands for improved performance of lubricating oil compositions spurs a constant search for new additives and lubricating oil formulations to achieve the desired goals. For example, an automatic transmission is a complex hydraulic mechanism which incorporates the functions of a torque converter, wet clutches and planetary gearing in a relatively compact sealed unit. This device requires a transmission fluid which provides lubricity, extreme pressure properties and carefully selected frictional properties. The fluid must not be corrosive to copper alloys or in any way deleterious to the synthetic seals in the transmission. An important requirement for the fluid is that it maintain good lubricity and friction modifying properties under the prolonged high-shear, high temperature conditions encountered in this environment.

Carboxylic acids or their derivatives are widely employed as lubricity agents or friction modifiers in automatic transmission fluids. These lubricity agents provide commercial lubricants having a useful service life. The lubricants, however, are definitely limited in their stability which directly reduces their useful life. This is indicated by a rising coefficient friction in the fluid and by the early onset of erratic or harsh shifting in service and in automatic transmission tests. Automobile manufacturers and lubricating oil formulators have been particularly interested in trying to overcome the unsatisfactory properties of current automatic transmission fluids.

2. Description of the Prior Art

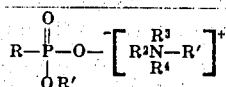
U.S. patent application, Ser. No. 778,335 filed on Nov. 22, 1968 discloses a lubricating oil composition containing an alkanephosphonic acid as a friction modifying agent.

U.S. patent application Ser. No. 812,410 filed on Apr. 1, 1970 discloses a lubricating oil composition containing a zinc, calcium or alkyl-ammonium acid salt of alkanephosphonic acid as a friction modifying agent.

SUMMARY OF THE INVENTION

A novel class of ammonium salts of alkyl alkanephosphonates has been discovered together with a method for preparing these compounds and a mineral lubricating oil composition which exhibits a variety of valuable properties.

The ammonium salts of alkyl alkanephosphonates are represented by the formula:



in which R is a substantially straight chain aliphatic radical having from about 11 to 40 carbon atoms, R' is a lower aliphatic radical having from one to eight carbon atoms, R² is a hydrocarbyl radical having from one to 40 carbon atoms and R³ and R⁴ are hydrogen, a hydrocarbyl radical having from one to 40 carbon atoms or

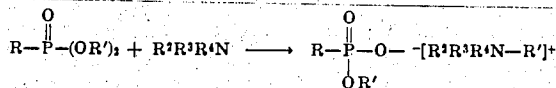
a substituted hydrocarbyl radical having amino, alkyl-amino or hydroxyl functional groups.

A preferred ammonium alkyl alkanephosphonate is one conforming to the above formula in which R is a predominantly straight chain aliphatic radical, i.e. one in which at least 75 percent and preferably 80 percent or more of the aliphatic carbon atoms are in a single straight chain. A completely straight chain aliphatic radical is suitable. Particularly preferred are those compounds in which R is a straight chain alkyl radical having from 12 to 18 carbon atoms. In the preferred ammonium alkyl alkanephosphonate, R' is an alkyl radical having from one to 2 carbon atoms, R² is an alkyl radical having from eight to 18 carbon atoms and R³ is hydrogen.

Examples of suitable ammonium alkyl alkanephosphonate additives of this invention include:

- | | |
|---|--|
| C ₁₄₋₁₅ sec. alkylmethylammonium methyl-n-tetradecylphosphonate, | |
| Dodecylmethylammonium | methyl-n-octadecylphosphonate, |
| Octadecylethylammonium | methyl-n-octadecylphosphonate, |
| Tetradecylbutylammonium | ethyl-n-hexadecylphosphonate, |
| Diocylammonium | methyl-n-dodecylphosphonate, |
| 3-(N-n-octadecylamino)propylmethylammonium | methyl-n-tetradecylphosphonate, |
| 3-(N-n-dodecylamino)propylethylammonium | methyl-n-dodecylphosphonate, |
| n-Butylmethylammonium | methyl-n-tetradecylphosphonate, |
| 3-Aminopropyl-n-octadecylmethylammonium | methyl-n-tetradecylphosphonate, |
| 3-Aminopropyl-n-dodecylmethylammonium | methyl-n-tetradecylphosphonate, |
| Dodecylmethylammonium | methyl-n-hexadecanephosphonate, |
| Dodecylmethylammonium | methyl-n-C ₁₂₋₁₈ alkylphosphonates, |
| n-Tetradecyltrimethylammonium | methyl-n-tetradecylphosphonate, |
| Di-n-dodecyltrimethylammonium | methyl-n-tetradecylphosphonate, |
| Di-mixed n-C ₁₀₋₁₄ sec. alkyltrimethylammonium | methyl-n-tetradecylphosphonate. |

The method of the invention involves the reaction of an alkanephosphonate diester with an amine according to the following formula:



in which R, R', R², R³ and R⁴ have the values noted above. This is a straightforward reaction and can be conducted at a moderately elevated temperature over a short reaction period. A temperature substantially greater than 80°C., e.g. 90°-130°C. is preferred while greater than 150°C. would usually be unnecessary. Reaction times of 2-5 hours are normally employed, although shorter times, e.g. 15 minutes to 1 hour, may be sufficient.

Catalysts are unnecessary and possibly would introduce undesirable impurities. Polar solvents, e.g. ethers, ketones, alcohols, might increase reaction rates. How-

ever if a solvent is desired, an inert, nonpolar solvent such as hydrocarbons are adequate provided the reaction is run at the preferred ranges of temperature and time. The amine may be used in substantial excess, e.g. from 1 to 2 moles of amine per mole of the alkanephosphonate diester without changing the reaction.

The following examples illustrate the method for preparing ammonium salts of alkyl alkanephosphonates.

EXAMPLE I

C₁₄₋₁₅ Sec. Alkylmethylammonium Methyl-n-tetradecylphosphonate

55 grams (0.255 mole) of C₁₄₋₁₅ sec. alkyl amine and 100 grams (0.255 mole) of dimethyl n-tetradecylphosphonate were added to a reaction vessel. The mixture was heated to a temperature in the range from about 115° to 125°C. and maintained under these reaction conditions for about 2.5 hours. Recovery of entire product was effected without further treatment or purification.

Analysis of the product was as follows:

	Found	Calculated
% Nitrogen	2.3	2.3
% Phosphorus	5.8	5.1
Total Acid No.	75	92 ^a

^aCalc. for one acidic H formed per phosphorus atom

EXAMPLE II

n-Dodecylmethylammonium Methyl-n-Tetradecylphosphonate

Forty-six grams (0.25 mole) of n-dodecylmonoamine (Armeen 12D) and 87 grams (0.25 mole) of dimethyl tetradecylphosphonate were mixed in a reaction vessel. The mixture was heated to a temperature in the range from about 115° to 125°C. and maintained under these conditions for 4 hours. On completion of the reaction the n-dodecylamine ammonium salt of monomethyl n-C₁₄ alkanephosphonate was recovered without further treatment, and analyzed as follows:

	Found	Calculated
% Nitrogen	2.5	2.6
% Phosphorus	6.6	5.8
Total Acid No.	98	105

EXAMPLE III

3-(N-n-octadecylamino)propylmethylammonium Methyl-n-Tetradecylphosphonate

One hundred grams (0.25 mole) of 3-(N-n-octadecylamino)-propylamine was dissolved in 100 ml. benzene and azeotroped free of water at ≈80°C. Then 75 grams (0.216 mole) of the dimethyl n-tetradecylphosphonate was added, and the solvent was distilled off, raising the temperature to 120°C. After 3 hours at 120°C., residual solvent was removed at 60°C. under 15 mm. of pressure. The phosphonate, a methylated n-octadecylamino-trimethyleneamine salt of monomethyl n-C₁₄ alkanephosphonate major product thus recovered had the following analysis:

	Found	Calculated
% Nitrogen	4.0	4.0
% Phosphorus	4.0	3.83
Total Acid No.	67	69

EXAMPLE IV

3-(N-n-Octadecylamino)propylmethylammonium Methyl-n-C₁₈ Alkanephosphonate

Forty grams (≈0.12 mole) of n-octadecylaminotrimethyleneamine dissolved in 100 ml. toluene and azeotroped to remove water. Then 43 grams (0.10 mole) of the dimethyl ester of n-C₁₈ alkanephosphonate was added and the mixture refluxed at 119°-122°C. for 3 hours. Solvent was removed by distilling to 100°C. under 14 mm. of pressure. The methylated n-octyldecylaminotrimethylene amine salt of the monomethyl ester of n-C₁₈ alkanephosphonate major product had the following analysis:

	Found	Calculated
% Nitrogen	3.8	3.4
% Phosphorus	3.9	3.7
Total Acid No.	69	68

An important feature of this invention is the discovery of a mineral lubricating oil composition which exhibits friction modifying properties making the lubricant particularly suitable for use in automatic transmission service. The lubricating oil composition comprises at least 86 weight percent of a mineral lubricating oil and from about 0.01 to 5.0 weight percent of an amine salt of the invention.

The automatic transmission fluid of the invention will generally be a fully formulated fluid containing minor amounts of conventional additives. For example, the automatic transmission fluid can contain from about 0.5 to 8.0 weight percent of a polymer of mixed alkyl esters of methacrylic acid having above 25,000 molecular weight, 0.25 to 5.0 weight percent of a reaction product of a polyalkylene polyamine and an alkenyl succinic acid, 0.1 to 5 weight percent of a zinc dialkylphenoxyalkyl dithiophosphate and from about 0.1 to 2.5 weight percent of an aryl-substituted alpha naphthylamine. The automatic transmission fluid of the invention is generally characterized by an SUS viscosity at 210°F. of 49 to 60, a viscosity index of at least 150 and a pour point below -40°F. and meets the essential DEXRON specifications set by General Motors for automatic transmission fluids.

The mineral lubricating oil which constitutes at least 86 weight percent of the composition of the invention is a refined oil or a mixture of refined oils selected according to the viscosity requirements of the particular service. For automatic transmissions where the requirements include an SUS viscosity of the compounded oil at 210°F. of 49 minimum up to 60 and at 0°F. of 7,000 maximum (extrapolated), the base oil or the major component thereof is generally a distillate oil lighter than SAE-10 grade motor oil, such as one having an SUS viscosity at 100°F. less than 150 and generally between about 50 and 125. The distillate fraction can be a refined paraffinic distillate, a refined naphthenic distillate or a combination thereof. The flash point of the distillate component of the base oil will generally be substantially above 300°F.; if the distillate fraction constitutes the entire base oil, its flash point will usually be above 375°F.

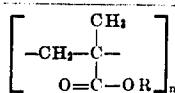
A particularly preferred base oil comprises approximately 70 to 95 percent of a refined distillate oil and 5 to 30 percent of a refined residual fraction which imparts desired high flash point and lubricity to the base

oil. A particularly preferred residual fraction comprises a paraffin base residuum which has been propane deasphalted and subjected to centrifuge dewaxing which has an SUS at 210°F. below about 250. An effective base oil mixture comprises 68 percent of a furfural re-
 5 refined, acid treated, clay contacted, solvent-dewaxed, paraffin base distillate having an SUS at 100°F. of 100, a viscosity index about 100, a flash above 385°F. and a pour below about +10°F., 22 percent of an acid-treated naphthenic base distillate having an SUS at
 10 100°F. of 60, a flash above 300°F., and a pour below -40°F., and 10 percent of a paraffin base residuum which has been propane deasphalted, centrifuge-dewaxed, and clay-contacted, and has an SUS viscosity at 210°F. of about 160, a flash above 530°F. and a pour
 15 of +5°F.

A surprising feature of the mineral lubricating oil composition of the invention is that it exhibits a marked reduction in friction between transmission clutch plates as the sliding speed approaches zero, a highly desirable
 20 feature for an automatic transmission fluid not possessed by lubricating oils containing carboxylic acids.

A relatively minor amount of the ammonium alkyl alkanephosphonate in a suitable lubricating oil is effective to provide an outstanding lubricating oil composition or an automatic transmission fluid. Generally, the salt is employed in the transmission fluid in an amount ranging from about 0.01 to 5.0 percent by weight with the preferred concentration of the alkane phosphonic
 25 acid being from 0.05 to 0.5 weight percent.

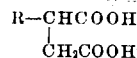
Viscosity index improvement of the fully formulated automatic transmission fluid of the invention is effected with a methacrylate ester polymer having the formula:



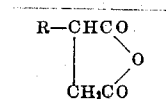
wherein R is an alkyl group, a dialkyl aminoalkyl group or a mixture of such groups containing from one to 20 carbon atoms and n is a member providing a molecular weight of the polymer in the range from 25,000 to 1,250,000 and preferably from 35,000 to 200,000. Various methacrylate ester polymers of this type are known which possess pour depressant and viscosity index improving properties. A very effective material of this type is a copolymer of the lower C_4 - C_{14} alkyl methacrylate esters. A commercial methacrylate copolymer of this type which is primarily a viscosity index improver corresponds to the formula in which R represents about 32 percent lauryl, 28 percent butyl, 26 percent stearyl and 14 percent hexyl groups and having a molecular weight above 50,000. The methacrylate ester copolymer is employed in the base oil in a proportion ranging from about 0.5 to 10 percent by weight preferably from 1.0 to 5.0 weight percent based upon the oil composition in order to impart the desired viscosity, viscosity index and pour point. It is understood that other methacrylate ester polymers of the foregoing
 65 type can be employed.

A dispersant is generally present in the automatic transmission fluid. An effective dispersant comprises a

composition resulting from mixing a substituted succinic compound, selected from the class consisting of substituted succinic acids having the formula:



and substituted succinic anhydrides having the formula:

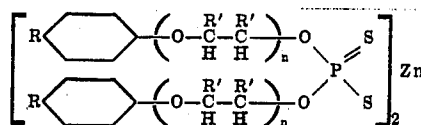


in which R is a large substantially aliphatic hydrocarbon radical having from about 50 to 200 carbon atoms with at least about one-half of a molar equivalent amount of a polyethylene polyamine and, in the case of the acid, heating the resultant mixture to effect acylation and remove the water formed thereby. The anhydride, however, may react without external heating and hence may be heated only if further reactions of the intermediate amic acid are desired. Suitable amines are diethylene triamine, triethylene tetramine, tetraethylene pentamine and amino-alkylated heterocyclic compounds. The reaction involves amidation of a dicarboxylic acid or anhydride thereof with a polymer to produce amino-substituted acyclic diamides, amic acids, polymeric amides, or a combination of these types of products. It will be noted that the amide groups may further react to form imide groups in the process.

Equivalents here means that a minimum of one-half mole of alkenylsuccinic anhydride or acid per mole of amine is required. This would be the least amount of acid which could react with all of the amine added (via amic acid or acyclic polyamide formation). The maximum amount of acid or anhydride possible to react is one-half mole per primary or secondary amino group. Generally, one or two moles of acid or anhydride per mole of amine, regardless of the total number of nitrogen atoms, is preferred. The reaction product is effective in amounts ranging from about 0.25 to 5.0 weight percent. Methods for preparing the polyethylene polyamine reaction products are well known and have been described in U.S. Pat. Nos. 3,131,150 and 3,172,892.

An amine anti-oxidant is important in this fully formulated transmission fluid. Effective anti-oxidants are the aryl-substituted amine anti-oxidants exemplified by the phenyl naphthyl amines, phenylene diamine, phenothiazine and diphenylamine. A particularly preferred antioxidant is phenyl alpha naphthylamine. The antioxidants are effective in a concentration ranging from about 0.1 to 2.5 weight percent.

A zinc di(alkylphenoxypolyalkoxyalkyl) dithiophosphate is a valuable component of a fully formulated automatic transmission fluid. This compound is represented by the formula:



in which R represents an aliphatic radical having from six to 15 carbon atoms, R' is hydrogen or an alkyl radical having from one to four carbon atoms and n is an integer from 1 to 10. Particularly effective compounds in this class are the zinc di(nonylphenoxyethyl) dithiophosphate, zinc di(dodecylphenoxyethyl) dithiophosphate and zinc di(nonylphenoxyethoxyethyl) dithiophosphate. Zinc di(nonylphenoxyethyl) dithiophosphate is prepared by reacting a nonylphenol-ethylene oxide compound with phosphorus pentasulfide followed by neutralization of the acid formed with a basic zinc compound, such as zinc carbonate, zinc oxide or zinc hydroxide. The general preparation of the compounds in this class is disclosed in U.S. Pat. No. 2,344,395 and 3,293,181. In use, it is convenient to prepare a mineral oil solution of the zinc di(C₆₋₁₅ alkylphenoxyalkoxyalkyl) dithiophosphate containing from 50 to 75 weight percent of the zinc salt. The salts are effective oxidation and corrosion inhibitors for automatic transmission fluids when employed in a concentration ranging from about 0.1 to 5.0 weight percent based on the hydraulic fluid.

Anti-foam agents are conventionally employed in hydraulic fluids because the fluids are rapidly circulated in operation and air can be entrapped. For this purpose, a silicone fluid of high viscosity, such as a dimethyl silicone polymer having a kinematic viscosity of 25°C. of about 1,000 centistokes and above is preferably employed. A very satisfactory anti-foam agent for this purpose is prepared by diluting 10 grams of a dimethyl silicone polymer (1,000 centistokes at 25°C.) with kerosene to provide a solution of 100 cubic centimeters. From 0.005 to 0.025 percent by weight of this concentrate is generally employed in the hydraulic fluid to provide from 50 to 200 parts per million of the silicone polymer based on the hydraulic fluid composition.

A fully formulated lubricating oil composition for automatic transmission service can be prepared from a base oil blend comprising 65 percent of a furfural-refined, acid-treated, clay-contacted, solvent-dewaxed, paraffin base distillate having an SUS at 100°F. of 100; a viscosity index about 100, a flash above 385°F. and a pour below +10°F., 22 percent of an acid-treated naphthenic base distillate having an SUS at 100°F. of 60, a flash above 300°F. and a pour below -40°F. and 13 percent of a paraffin base residuum which has been propane-deasphalted, solvent-dewaxed and clay-contacted and which had an SUS viscosity at 210°F. of 160, a flash of about 540°F. and a pour below 5°F. This base oil mixture had a flash above 375°F., a pour below 0°F. and a viscosity index of about 93.

A fully formulated automatic transmission fluid will comprise a base oil blend such as the foregoing and will contain from about 0.5 to 8 weight percent of an oil concentrate containing about 35 percent of a basic amino nitrogen-containing addition type copolymer comprising copolymers of butyl, lauryl, stearyl and dimethyl aminoethyl methacrylates in approximately 21:53:22:4 weight ratios (as described in U.S. Pat. No. 2,737,496); about 0.25 to 5.0 weight percent of an oil concentrate containing about 33 percent of the reaction product of approximately 1:1 mole ratio of tetraethylene pentamine and alkenyl succinic anhydride in which the alkenyl radical is polybutene of approximately 1200 average molecular weight (U.S. Pat. No. 3,172,892); about 0.1 to 2.5 weight percent of phenyl

alpha naphthylamine, about 0.1 to 5 weight percent of an oil concentrate containing about 50 percent of zinc di(nonyl-phenoxyethyl) dithiophosphate and from about 0.01 to 5 weight percent of the ammonium alkyl alkanephosphonate of the invention.

The frictional effects of the ammonium alkyl alkanephosphonate additive of the invention on a mineral lubricating oil was determined in a Low Velocity Friction Test. This test was conducted using a General Motors type friction surface, namely a test plate identical in composition to Borg Warner's SD-715 clutch plates, in sliding contact with steel. The coefficients of friction were determined at decreasing sliding speeds, i.e. from about 40 ft./min. down to about 1 ft./min. The test temperature was 250°F. and the applied load was 120 psi.

The frictional effects of various ammonium alkyl alkanephosphonates was determined in the Low Velocity Friction Test. The base oils employed were paraffin base mineral oils that had been solvent refined and solvent dewaxed. Base Oil A had an SUS viscosity at 100°F. of about 100 and Base Oil B had an SUS viscosity at 100°F. of about 340. The coefficients of friction for the various blends at different sliding velocities are set forth in the table below:

TABLE I

Run	Base Oil + Wt. % of Ammonium salt of monoalkyl alkanephosphonate	Coefficient of Friction Sliding Velocity, ft./min.	
		40 ft./min.	Approximately 1.0 ft./min.
1.	Base Oil A	0.119	>0.180
2.	Base Oil A + 0.26 wt. % Example I	0.105	0.062
3.	Base Oil A + 0.21 wt. % n-Butyl Methylammonium methyl-n-tetradecyl-phosphonate	0.104	0.057
4.	Base Oil B	0.085	0.140
5.	Base Oil B + 0.25 wt. % Example II	0.089	0.043
6.	Base Oil B + 0.38 wt. % Example III	0.100	0.055

Runs 2, 3, 5 and 6 show that the coefficient of friction of these blends was sharply reduced as the sliding velocity approached 1.0, a particularly valuable property for smooth shifting in automatic transmissions. In contrast, the coefficient of friction of Base Oils A and B alone increased greatly as the sliding velocity decreased showing that these oils in the absence of the additive are unsatisfactory.

Oil blends were prepared to compare a fully formulated automatic transmission oil composition of the invention with a similar base blend and a blend containing a commercial friction modifier additive. These fluids were tested for the length of their Friction Limited Life and for the period of time that the fluids exhibited Smooth Up-Shift characteristics in the standard GM

Dexron Power-glide T-12 Cycling Test. The oil blends had the following compositions:

TABLE II

	Blend A	Blend B	Blend C
1. Paraffin base distillate having SUS at 100°F. of 100	56.25	58.05	60.0
2. Naphthenic base distillate having SUS at 100°F. of 60	20.0	20.0	20.0
3. Paraffin base residuum having an SUS at 210°F. of 160	14.0	12.0	10.0
4. VanLube NA ¹	0.60	0.60	0.60
5. Zinc dialkylphenoxy ethyldithiophosphate (68% solution in oil)	1.25	1.25	1.25
6. 1:1 mole ratio reaction product of tetraethylene pentamine and polybutene-1200 succinic anhydride (25% oil solution)	3.90	3.90	3.90
7. Butyl, lauryl, stearyl and dimethylaminomethacrylates blend in 21:53:22:4 weight ratios (35% in oil)	4.00	4.00	4.00
8. Sarcosyl S ²	—	0.20	—
9. Additive of Example III	—	—	0.25

¹VanLube NA=essentially about 2/3 diethyl-tert.-dioctyl-diphenylamine and 1/3 diethyl-tert.-monoocetyldihexylamine.
²Sarcosyl S=stearyl sarcosine, commercial friction modifier.

The test results obtained by running the above fluids in the T-12 Cycling Test are set out in the Table below.

TABLE III

Dexron Powerglide T-12 Cycling Test

	Friction Limited Life (hours)	Smooth Up-shift Life (hours)
Blend A	229	0
Blend B	232	67
Blend C	356	200

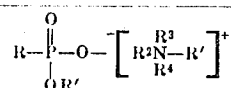
Blend C which is a formulated automatic transmission fluid according to the invention is outstanding in both Friction Limited Life and Smooth Up-Shift Life in contrast to a similar fluid containing a commercial fric-

tion modifying agent.

The novel compounds of the invention as well as lubricating oil compositions containing these compounds have been shown to possess outstandingly valuable properties when used in lubricating oil compositions.

I claim:

1. A lubricating oil composition comprising at least 86 weight percent of a mineral lubricating oil and from about 0.01 to 5.0 weight percent of an ammonium alkyl alkanephosphonate represented by the formula:



in which R is an aliphatic radical having from about 11 to 40 carbon atoms and in which at least 75 percent of the carbon atoms are in a straight chain, R' is a lower aliphatic radical having from one to eight carbon atoms, R² is a hydrocarbyl radical having from one to 40 carbon atoms and R³ and R⁴ are hydrogen, or a hydrocarbyl radical having from 1 to 40 carbon atoms.

2. A lubricating oil composition according to claim 1 in which R is a hydrocarbon radical in which 80 percent of the carbon atoms are in a straight chain.

3. A lubricating oil composition according to claim 1 in which R is a straight chain aliphatic hydrocarbon radical.

4. A lubricating oil composition according to claim 1 in which said ammonium alkyl alkanephosphonate is C₁₄₋₁₅ sec. alkylmethylammonium methyl-n-tetradecylphosphonate.

5. A lubricating oil composition according to claim 1 in which said ammonium alkyl alkanephosphonate is n-dodecylmethylammonium methyl-n-tetradecylphosphonate.

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